

Temporal Stability of Oxidizer Solutions: A Survey of Candidate Decon Materials in Seawater

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TEMPORAL STABILITY OF OXIDIZER SOLUTIONS: A SURVEY OF CANDIDATE DECON MATERIALS IN SEAWATER

INTRODUCTION

Naval operations may be compromised by the presence of Chemical Warfare/Biological Warfare (CW/BW) agents and/or toxins. In such an environment, the Fleet's effective operational status can be maintained only if the U. S. Navy has dependable, readily implementable decontamination (decon) procedures. A key element in this CW defense posture is the ability, on an as-needed basis, to prepare and apply chemically active decon fluids to counter the CW/BW threat.

The majority of CW agents/toxins will succumb to a chemically active decon utilizing oxidation/hydrolysis. The present active decon of choice relies on hypochlorite ion, which in aqueous solution provides both hydrolytic and oxidative destruction of G-agents (Block and Davis, 1978). At pH less than 7, hypochlorite (at this pH, predominantly hypochlorous acid) is effective in neutralizing BW materials such as spores (Hoffman and Spiner, 1962; Fielding, et al., 1967, 1968). Thus, decon with aqueous hypochlorite has the potential to be both rapid, and broadly effective against both CW and BW materials. Historically, calcium hypochlorite has been the preferred source of hypochlorite ion.

However, serious questions and limitations exist concerning the overall utility of a decon procedure requiring calcium hypochlorite. Calcium hypochlorite exhibits instability in storage, disperses slowly in water to give a solution/slurry which can be troublesome to dispense mechanically, suffers from handling and logistics problems as a bulk solid. Needed, then, are data which would allow the choice of potential supplements or replacements for calcium hypochlorite. These candidate alternative decon materials should possess the broad effectiveness of hypochlorite against both CW and BW materials, exhibit improved stability in storage, and for Naval applications be compatible for use with seawater.

Utilization of decon fluids prepared with seawater is of vital interest to the Navy, given the Navy's unlimited access to seawater. A seawater-based decon can be utilized for the decon of large surface areas such as decks, bulkheads, and materials storage areas. Yet, a seawater-based decon technology requires candidate decon reagents stable both in storage and in solution.

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A recent report (Pellenbarg, 1985) examined the temporal stability of solutions of candidate oxidizers in distilled water. These data provide some insight into the storage stability of solutions of various candidate solid oxidizers. However, the earlier investigation did not address the stability of candidate oxidizer solutions in seawater. This report provides preliminary seawater solution stability data for some candidate oxidizers. Thus, preliminary information concerning the stability of solid candidate oxidizers dissolved in seawater, as well as fresh water, is now available, and can contribute to preserving an effective operational posture of the U.S. Navy.

EXPERIMENTAL

Decon associated with Naval operations would likely, in part, rely on seawater, given its availability. The following procedure was established to provide information on seawater solution stability of candidate decon materials. The candidate chemicals were dissolved in seawater, and stored as solutions at cold (6°C), ambient (23°C), and hot (~70°C) temperatures. Periodically, aliquots of these three groups of samples were iodometrically titrated to assess residual oxidizer content of the solutions as a function of storage time. Table 1 lists the oxidizers tested.

For the current study, all test solutions were prepared in full strength seawater (34.3 o/oo salinity, from the North Atlantic at approximately 53°N, 36°W; Table 2 lists the typical composition of seawater). Hypochlorite solutions were made up to 0.1 N concentration (Fielding, et al., 1967, 1968). All hypochlorite reagents were used as packaged, to resemble what could occur in the field. The solid hypochlorites (calcium, lithium, and PACE) were weighed out for addition to seawater, and a commercial sodium hypochlorite solution was diluted as required with seawater.

In contrast to the hypochlorite solutions, the peroxygen solutions were made up to a lower concentration (0.01 to 0.02 N). The solid peroxygen salts, especially sodium peroxydisulfate and sodium perborate, required a long time to dissolve. The lower concentration was selected as a compromise between higher concentration test solutions and the time required to prepare the solutions. Note that this time constraint has important implications for field use. The required hydrogen peroxide solutions were diluted with seawater from a concentrated commercial stock.

Aliquots (200 ml) of the unbuffered test solutions of hypochlorites and peroxygen reagents were divided into three groups. One group of solutions in Pyrex beakers was covered loosely with Parafilm, and held in a refrigerator at 6°C. A second group was stored loosely covered at ambient temperature, ~23°C. The third group was retained at ~72°C. These high

temperature samples were placed in a Soxhlet extraction apparatus fitted with straight condensers to minimize evaporation of water from the test samples. This procedure prevented concentration changes through evaporation.

The oxidizer content of the seawater solutions was monitored by iodometric titration. Iodometry relies on the oxidation of iodide ion to free iodine, which then is quantitatively determined by titration with aqueous thiosulfate. Using hypochlorite as an example, (1) and (2) pertain:



For the iodometric titrations, 2.0 ml aliquots of test solution were taken in triplicate as a function of time, and dispensed into separate 200 ml beakers containing 100 ml distilled water. The pH of the solution was adjusted to $\sim 4.5 \pm 0.5$ with 2 N H_2SO_4 ; in distilled water, then 4.0 ml of 10% wt/wt potassium iodide in distilled water was added to the titration beaker with stirring. The resulting solution was titrated to a pale straw color with 0.1 N sodium thiosulfate reagent (Fisher), ~20 drops of starch dissolved in water were added to give a dark blue solution, and the sample titrated to a clear, colorless endpoint with additional thiosulfate. The total volume (titer) of thiosulfate reagent needed to reach the endpoint was recorded. These titer data, as well as the test solution pH, both as a function of time, are listed in Tables 3 and 4. Process blanks, consisting of 100 ml distilled water, 2.0 ml seawater, and necessary reagents, consumed no thiosulfate, consistently indicating low to zero background oxidants in the house distilled water and reagents used in this study. All pH measurements were performed with a Fisher Accumet Model 300 pH meter and micro combination pH electrode.

The candidate oxidizers exhibited differences in their reaction rates with iodide. For the hypochlorite salts, and H-48, the oxidation of the iodide was complete as soon as the reagents were mixed. However, several of the other peroxygen compounds oxidized the iodide more slowly. To better accommodate the slower peroxygen reaction kinetics, the hydrogen peroxide-iodide solution was allowed to react for 10 minutes, and the perborate-iodide and peroxydisulfate-iodide interactions were allowed to proceed for 20 minutes prior to titration with thiosulfate. These arbitrary reaction times served as a compromise between seeking complete reaction between peroxygen anion and iodide, and being able to complete the required titrations in a timely manner.

RESULTS AND DISCUSSION

Tables 3 and 4 list the titers of thiosulfate solution equivalent to the quantity of residual oxidizer in the various solutions tested. For this study, the residual oxidizer was monitored as a function of two major variables, namely time and temperature. Results related to these two variables will be discussed separately.

Temporal Stability

All of the inorganic hypochlorite salts exhibited good long term stability in seawater (see Figure 1), except at high temperature, as will be discussed later. The observed low temperature stability is in contrast to what was observed in fresh water (Pellenbarg, 1985). In fresh water, calcium hypochlorite was unstable with time at ambient temperature. This was not the case in seawater. It is hypothesized that the mix and concentration of cations in seawater (Table 2) stabilizes calcium hypochlorite in the following manner: For calcium hypochlorite, in fresh water solution, the positive calcium ion can precipitate with time as calcium hydroxide or carbonate, leaving a less stable solution of hypochlorous acid. Seawater, however, contains a mix of positive ions, especially sodium, not subject to precipitation reactions. These very soluble cations could serve to stabilize the "calcium" hypochlorite solution. Thus, even if calcium ion precipitated, other cations present would preclude the formation of less stable hypochlorous acid. This aspect of hypochlorite stability in seawater (i.e., a common ion effect) needs to be investigated in more detail, with special attention to the effects of magnesium ion on hypochlorite stability.

The stability of the "organic" based hypochlorite tested was quite different than that observed for the inorganic hypochlorite salts just discussed. Sodium dichloroisocyanurate [$\text{Cl}_2\text{Na}(\text{NCO})_3 \cdot 2\text{H}_2\text{O}$, PACE, Olin Corporation] was examined in the current investigation as a candidate decon substance. In seawater, PACE was stable for several days at low temperature, but lacked stability at higher temperatures (23°C , 72°C). It is possible that the organic moiety (cyanuric acid) produced when PACE decomposes (Figure 2) to yield hypochlorous acid may partially consume the released hypochlorite, causing the observed deterioration of solutions of PACE in seawater. This hypothesis can be examined by monitoring the appearance of cyanuric acid oxidation products such as formic acid, carbon dioxide, and/or chloramines. These measurements were beyond the scope of the current investigation.

As a class, the examined peroxygen compounds, like the inorganic hypochlorite salts, exhibited similar stabilities in seawater, with the exception of H-48 (an "organic" peroxygen reagent, see Table 3). At all the temperatures studied, solutions of H-48 in seawater displayed severe loss of oxidizer

capacity in only a few hours. This result is in contrast to that reported earlier (Pellenbarg, 1985) when H-48 was observed to be stable for up to 24 hours in distilled water. Again, seawater as an ionic medium may catalyze the decomposition of H-48 to an organic residue and peroxide ion. The peroxide would be consumed by interaction with the residual organic matter, contributing to the observed instability of the H-48 solutions. This process might be similar to that proposed for the observed instability of PACE solutions. The detailed chemistry of both H-48 and PACE in seawater should be investigated, with special emphasis on the effects of trace metals (copper, iron, cobalt, zinc, nickel) on the stability of oxidizer solutions. Traces of iron are known to catalyze the decomposition of hydrogen peroxide solutions, for example (Cotton and Wilkinson, 1966).

All of the inorganic peroxygen salts studied exhibited similar stability with time in seawater. This stability may be related to the absence of an oxidizable organic moiety in the test solutions. This hypothesis could be validated by adding a soluble, easily oxidized organic material to the inorganic peroxygen solutions. Such interactions should be examined, for two reasons. First, natural seawater contains some 3-4 mg/L (Duursma and Dawson, 1981) dissolved organic matter. This organic matter could interact with and consume added oxidizers, reducing the effectiveness of a decon fluid made up in seawater. Second, a particular decon formulation may require the addition of various organic additives (eg., a detergent) to a particular decon formulation for efficacy. If such additives could consume the oxidizer(s) in the decon fluid, this possibility must be known. Future work should examine the synergistic or antagonistic interactions of oxidizers/hydrolyzers and organic additives, such as Aqueous Film Forming Foam, in recommended decon formulations.

The pH of the solutions tested fell into two major groupings. The hypochlorite salt solutions were distinctly alkaline ($\text{pH} > 8$), except for the PACE solution which was mildly acidic ($\text{pH} \sim 6.5$). The peroxygen solutions were slightly alkaline ($\text{pH} \sim 7.5$) to distinctly acid. The various candidate reagents tested interacted with the natural buffering capacity of seawater (pH of open ocean seawater is $\sim 8.1\text{-}8.3$, alkalinity = $2[\text{CO}_3^{2-}] + [\text{HCO}_3^{-}] + [\text{B(OH)}_4^{-}] + [\text{OH}^-] - [\text{H}^+] \approx 4 \text{ mM}$; Riley and Chester, 1971). All of the peroxygen compounds overwhelm the buffering capacity of the seawater used in these tests, while the hypochlorite salts did not greatly affect the original pH of the seawater. These pH adjustment processes are important from an user point of view, as pH of the solutions applied for decon could affect both the efficacy of the decon solution, and its corrosivity. Both these pH related effects should be examined in detail, in a seawater matrix, using appropriate CW/BW simulants, and Naval alloys.

Thermal Stability

The second major variable in the current investigation was solution temperature. Solution stability tests were replicated at 6°C, 23°C, and 72°C. In general, the hypochlorite salt solutions were most stable at the low and intermediate temperatures, and less stable at high temperature (see Figure 1). Hypochlorite decomposition reactions proceed more rapidly at the higher temperatures. Thus, in a tactical decon context, hypochlorite is unstable in seawater at high temperature, and high temperatures may impact adversely on solid hypochlorite in storage.

All peroxygen solutions tested were more stable at the lower temperatures, as was observed with the hypochlorite solutions. Of the peroxygen compounds examined, perborate and peroxydisulfate exhibited stability for approximately 24 hours at the highest temperature examined (-70°C). Hydrogen peroxide and H-48 were stable for only a couple of hours at this higher temperature. In all cases, decomposition reactions are accelerated at elevated temperatures. This phenomenon dictates that one use caution when considering the use of high temperature seawater-based decon fluids containing peroxygen compounds. Overall, the current data indicate that it may be unwise to store decon solutions, especially at high temperatures ($T > 30^\circ\text{C}$), for long periods prior to use. Also, the user must recognize the potential for decomposition of solid peroxygen materials and hypochlorites, stored at higher temperatures. Needed are data describing the temporal stability of the solid candidate decon materials as a function of storage temperature (especially $T > 30^\circ\text{C}$).

CONCLUSIONS

In the preliminary experiments described in this report, two classes of oxidizers were examined. The stability of these oxidizers in seawater solution were measured as functions of time and temperature. All of the inorganic hypochlorites were acceptably stable in seawater solution at low and ambient (6°C to 23°C) temperature. This stability is ascribed to the overall ionic composition of seawater. Potential decomposition reactions related to the cation in the hypochlorite salt are not observed in seawater because of the high concentration and variety of cations present. However at the higher temperatures ($> 70^\circ\text{C}$) no hypochlorite in seawater was stable for more than 24 hours. If hypochlorite solutions in seawater are to be made prior to need from solid starting materials, these solutions should be kept cool to maximize their stability.

The current study dealt with the stability in seawater of one organic-based hypochlorite reagent. This compound, sodium dichloroisocyanurate (PACE) was acceptably stable at the low and ambient temperatures, but was stable for only some 3-4 hours at

a higher temperature. A mechanism is postulated in which the liberated hypochlorite is consumed by oxidation of the organic residue resulting from the initial release of the hypochlorite. From a solution stability viewpoint, PACE may prove to be an acceptable starting material for a hypochlorite decon fluid, but its efficacy against CW/BW simulants/agents remains to be demonstrated.

Of the peroxygen compounds tested, perborate and peroxide appeared acceptably stable in solution at all temperatures, and for times up to 24 hours. Peroxydisulfate decomposed rapidly at high temperature, and H-48 was unstable in unbuffered seawater at all temperatures for periods of more than about 0.5 hour. All the peroxygen solutions reacted slowly with iodide during titration, as was observed for tests conducted in fresh water. These reaction kinetics, and indeed the peroxygen stabilities observed, may be pH related (unbuffered peroxygen test solutions were neutral to acidic), and could affect overall efficacy of a peroxide based decon. Also, sodium perborate solutions required an inordinate amount of time to prepare. This time factor has tactical implications for decon operations.

RECOMMENDATIONS

This preliminary study of seawater based oxidizer solutions has identified several factors which can impact directly on the utility of aqueous oxidizers for Naval decon applications. Specifically needed are data which:

- (1). Compare the oxidation/hydrolysis (decon) ability of candidate compounds with appropriate CW/BW simulants and, ultimately, agents/toxins, in a seawater matrix, which is of special concern to the Navy.
- (2). Investigate the corrosivity to Naval alloys and materials associated with solutions of various peroxygen and hypochlorite species in unbuffered seawater, especially at the pH resulting from mixing these solids with seawater.
- (3). Examine the stability, as functions of temperature and humidity, of the bulk solid reagents used in the tests described in this study.
- (4). Study the potential interactions of candidate decon reagents, and organic additives such as detergents to determine that such interactions will not adversely affect the efficacy of the active decon reagents.

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Table 1 - Oxidizers Tested

Name	Formula wt	Calculated Oxidizer Normality	Salt g/l
Sodium Hypochlorite ^a NaOCl	74.5	0.1173	83 ml diluted to 1 l
Lithium Hypochlorite ^b LiOCl	58.4	0.08990	6.5630
Calcium Hypochlorite ^a Ca(OCl) ₂	143.1	0.1002	3.5843
Sodium Dichloroisocyanurate ^c dihydrate Cl ₂ Na(NCO) ₃ ·2H ₂ O (PACE)	255.0	0.1002	6.4120
Sodium Perborated ^d NaBO ₃ ·4H ₂ O	153.8	0.0202	1.5511
Sodium Peroxydisulfated ^d Na ₂ S ₂ O ₈	238.0	0.0199	2.3725
Hydrogen Peroxide ^a H ₂ O ₂	34.0	0.012 N	10 ml diluted to 1 l
Magnesium Bis (2-Carboxylato mono peroxybenzoyl acid) hexahydrate (H-48) Mg C ₁₆ H ₂₄ O ₁₆	496.3	0.0200	4.9582

^a The Clorox Company, Oakland, CA^b Lithium Corporation of America, Bessemer City, NC^c Olin Corporation, Cheshire, CT^d Alfa Products, Danvers, MA^e Interox America, Houston, TX

Table 2 - Composition of Seawater

<u>Ion</u>	<u>Concentration*</u>	<u>Molarity</u>
Chloride, Cl ⁻	19.35	0.55
Sodium, Na ⁺	10.76	0.47
Sulfate, SO ₄ ⁼	2.71	0.034
Magnesium, Mg ⁺⁺	1.29	0.053
Calcium, Ca ⁺⁺	0.41	0.010
Potassium, K ⁺	0.40	0.010
Bromide, Br ⁻	0.07	0.001
Strontium, Sr ⁺⁺	0.007	0.0001
Boron, B(OH) ₄ ⁻	0.004	0.0004
Fluoride, F ⁻	0.001	0.0001

* grams ion per kg of seawater of 35 0/00 (parts per thousand) salinity

Table 3 - Data for Hypochlorite Salts Tested

1. Lithium Hypochlorite (LiOCl)

	<u>time (hrs)</u>	<u>pH</u>	<u>titer (mL)</u>
A. cold	0	9.2	0.89
	26	8.8	0.84
	48	8.7	0.78
	144	8.5	0.67
	167	8.4	0.65
B. ambient	0	9.2	0.90
	3	8.8	0.87
	25	9.7	0.67
	47	8.6	0.64
	141	8.5	0.64
	165	8.5	0.64
C. hot	0	9.2	0.90
	2	8.4	0.63
	24	8.2	0.51
	44	8.1	0.44
	139	7.9	0.19
	163	7.9	0.17

2. Calcium Hypochlorite [Ca(OCl)₂]

	<u>time (hrs)</u>	<u>pH</u>	<u>titer (mL)</u>
A. cold	0	9.6	1.32
	26	8.9	1.33
	48	8.9	1.32
	144	8.5	1.31
	160	8.5	1.32
B. ambient	0	9.6	1.32
	3	9.5	1.31
	25	9.4	1.30
	46	9.4	1.27
	141	9.2	1.14
	165	9.1	1.14
C. hot	0	9.6	1.32
	2	8.7	1.21
	21	8.4	0.94
	45	8.2	0.74
	139	7.6	0.24
	163	7.6	0.19

Table 3 - (continued)

3. Sodium Hypochlorite (NaOCl)

	<u>time (hrs)</u>	<u>pH</u>	<u>titer (mL)</u>
A. cold	0	9.2	2.34
	26	9.0	2.30
	48	8.8	2.23
	144	8.8	2.22
	168	8.8	2.21
B. ambient	0	9.2	2.34
	3	9.2	2.30
	25	9.1	2.20
	47	9.2	2.20
	141	8.8	2.20
	165	8.9	2.16
C. hot	0	9.2	2.34
	2	8.6	2.17
	24	8.1	0.63
	44	7.9	0.33
	139	7.8	0.12
	164	7.7	0.11

4. PACE [Cl₂Na(NCO)₂·2H₂O]

	<u>time (hrs)</u>	<u>pH</u>	<u>titer (mL)</u>
A. cold	0	6.6	1.90
	26	6.3	1.90
	48	6.2	1.83
	144	5.7	1.69
	167	5.8	1.65
B. ambient	0	6.6	1.90
	3	6.2	1.90
	24	5.3	1.63
	47	4.9	1.43
	143	3.1	0.79
	166	2.8	0.69
C. hot	0	6.6	1.90
	2	3.8	1.00
	19	1.9	0.09
	44	1.9	0.03
	140	1.8	0
	164	1.8	0

Table 4 - Data for Peroxygen Salts Tested

1. Sodium Perborate (NaBO₃)

	<u>time (hrs)</u>	<u>pH</u>	<u>titer (mL)</u>
A. cold	0	5.3	0.10
	23	5.3	0.07
	46	5.3	0.11
	117	5.2	0.09
	164	5.0	0.07
B. ambient	0	5.3	0.10
	27	5.3	0.07
	45	5.3	0.08
	117	9.3	0.05
	163	9.2	0.06
C. hot	0	5.3	0.10
	23	3.9	0.07
	45	6.2	0.07
	117	8.1	0.04
	162	7.7	0.04

2. Sodium Peroxydisulfate (Na₂S₂O₈)

	<u>time (hrs)</u>	<u>pH</u>	<u>titer (mL)</u>
A. cold	0	7.7	0.03
	24	1.8	0.02
	47	7.7	0.01
	119	7.6	0.01
	166	7.6	0.01
B. ambient	0	7.7	0.03
	24	7.6	0.02
	48	7.6	0.01
	118	7.4	0.02
	164	7.4	0.03
C. hot	0	7.7	0.03
	21	3.9	0.03
	46	2.0	0.01
	117	2.2	0
	163	2.1	0

Table 4 - (continued)

3. H-48 (MgC₁₆H₂₂O₁₆)

	<u>time (hrs)</u>	<u>pH</u>	<u>titer (mL)</u>
A. cold	0	4.1	0.48
	2	-	0.06
	23	4.1	0
B. ambient	0	4.1	0.48
	10 min		0.32
	20 min		0.23
	35 min		0.14
	1		0.07
	1.5		0.03
	3		0.02
	24	4.0	0
C. hot	0	4.1	0.48
	2	-	0

4. Hydrogen Peroxide (H₂O₂)

	<u>time (hrs)</u>	<u>pH</u>	<u>titer (mL)</u>
A. cold	0	7.8	0.40
	27	7.6	0.47
	47	7.7	0.47
	118	7.6	0.31
	166	7.5	0.27
B. ambient	0	7.8	0.40
	26	7.6	0.56
	47	7.7	0.27
	118	7.8	0.09
	166	8.0	0.04
C. hot	0	7.8	0.40
	24	6.2	0

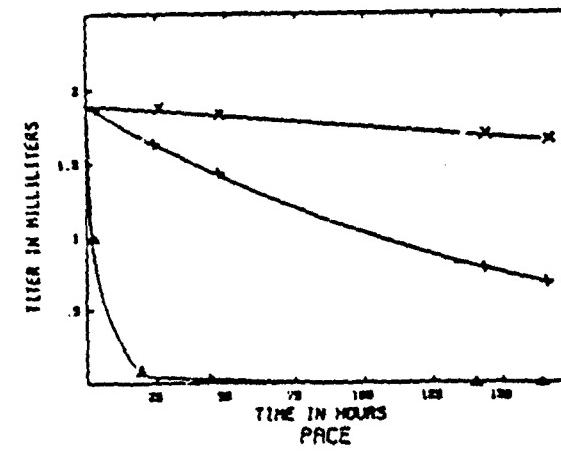
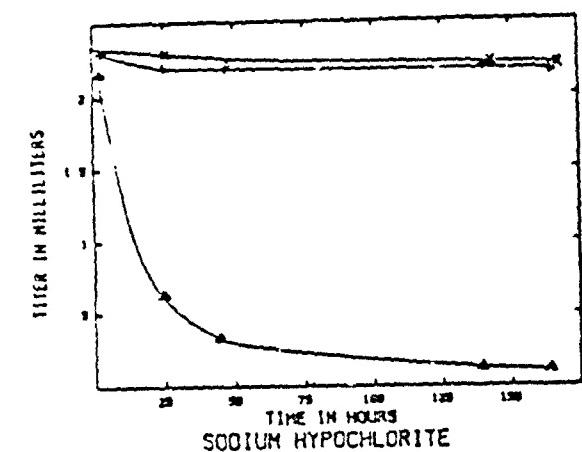
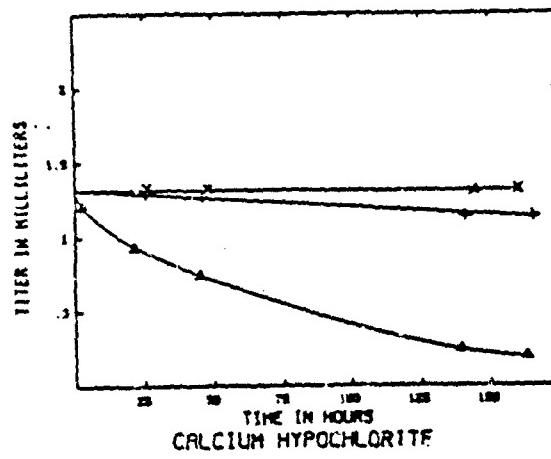
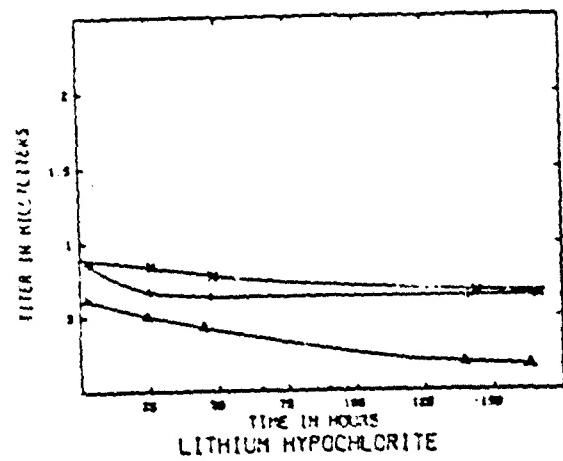


FIGURE I: Graphical presentation of data from chlorine-based oxidants. X is cold temperature, + is ambient, ▲ is hot.

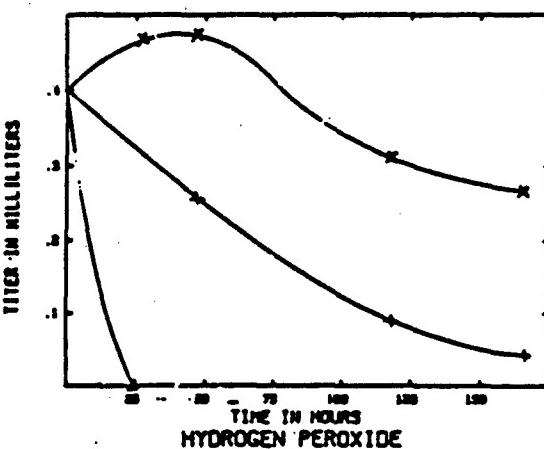
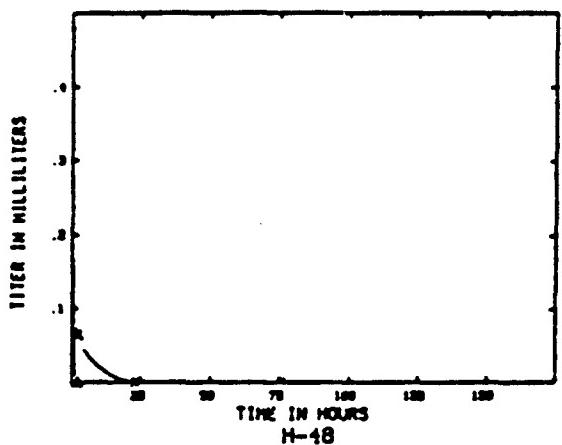
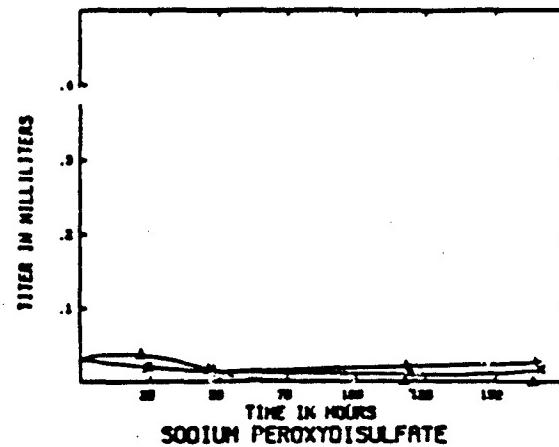
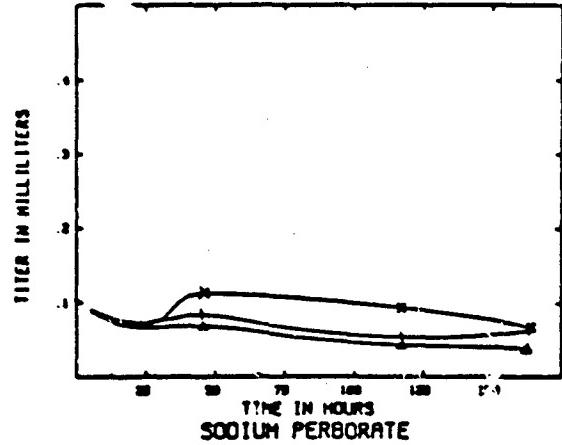


FIGURE 2: Graphical presentation of data from oxygen-based oxidants. X is cold temperature, + is ambient, ▲ is hot.